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(54) Method of preparation of silver halide emulsions for colour photographic materials

The present invention relates to a method of preparation of silver halide emulsion for colour photographic materials, for which the required viscosity is not achieved by dilution with water, but by addition of suitable substances, which makes it possible to increase the pouring rate for emulsions for which the glazing rate is limited by the capacity of the dryer, and improve the efficiency of the manufacturing technology.

Silver halide emulsions for colour photographic materials usually contain non-diffusing colour-forming components, which have one or more acid functional groups in their molecule (especially carboxy, sulpho or methylenic with a detachable hydrogen) which increases their solubility in an aqueous medium. Restriction of the diffusion of the colour-forming components is achieved in that the molecule of the colour-forming component contains an aliphatic chain, which prevents the components used in the gelatin layers from diffusing between these layers. Of course, these substituents endow the colour-forming components with properties of surface-active substances and greatly affect their binding capacity, which for the most part is utilised in photographic emulsion during their interaction with gelatin. The consequence of this interaction is the formation of a component-gelatin complex, which is manifested as a considerable increase in viscosity of emulsion containing a colour-forming component with an acid functional group.

The usual types of production equipment for glazes of colour photographic emulsions (wetting and extrusion) do not allow a good-quality glaze to be produced at high emulsion viscosities. Regardless of the absolute values, for all the emulsions used it is in addition necessary to adjust the viscosity of the emulsion prepared for glazing, because this governs the flow rates, and possibly the amount of emulsion deposited on the substrate. A common, and the most used, method of adjusting the viscosity of emulsion prepared for glazing is to make use of its dependence on concentration, i.e. dilution with water. Of course, an increased content of water in the emulsion means higher energy consumption for its evaporation in the dryer. Another adverse consequence of this usual method is that it is not possible to increase the pouring rate

for the emulsion, when the rate of glazing is limited by the capacity of the dryer, owing to high water content in the emulsion.

The aforementioned drawbacks are overcome by a novel method of preparation of silver halide emulsions for colour photographic materials, characterised in that a salt of an alkaline earth is added at the rate of 0.02 to 2 g/1 g of colour-forming component at a temperature in the range 20-80°C either to a solution of the colour-forming component with acid functional groups before it is incorporated in the emulsion, or to a photographic emulsion containing gelatin and a colour-forming component with acid functional groups. This results in a decrease in emulsion viscosity depending on the chosen dose of alkaline earth salt, at best to values that would be displayed by the emulsion without the colour-forming component. The amount of alkaline earth salt is determined by the stoichiometric proportions (the molecular weight and number of acid functional groups in the molecule of the colour-forming component and the molecular weight of the alkaline earth salt used) and the required decrease in viscosity. The dependence of viscosity on the amount of alkaline earth salt added is continuous, so that by selecting the dose it is possible to choose the degree of reduction of emulsion viscosity.

On the basis of this invention, components that increase the emulsion viscosity to unacceptably high values even at low concentration can be applied industrially. Moreover, using this invention it is possible to reduce the amount of water in emulsions containing gelatin and a colour-forming component with acid functional groups and the increase in viscosity caused by the increase in concentration can be counteracted with the additive according to the invention. In this way considerable energy savings can be made during drying of the poured material. Using this invention, it is also possible to achieve an increase in pouring rates for emulsions for which the glazing rate is limited by the capacity of the dryer owing to excessively high water content, thereby increasing the capacity of the whole plant.

Example 1

Add 0.1 to 10 g of $MgSO_4 \cdot 7 H_2O$, or a corresponding amount of arbitrarily concentrated aqueous solution of this salt, to 125 ml of water in which 5 g of component I has been dissolved with the aid of 10 ml of alcohol and 20 ml of 1N NaOH. The aqueous-alkaline solution of colour-forming component prepared in this way is used for preparation of emulsion for colour photographic materials by the usual method.

Example 2

In any stage of preparation of emulsion for glazing, but preferably after incorporating the solution of colour-forming component in the emulsion, 0.02 to 2 g of $MgSO_4 \cdot 7 H_2O$ or a corresponding amount of its aqueous solution is added, per 1 g of the colour-forming component used, at a temperature of 35 to 50°C, to a photographic emulsion prepared in the usual way and containing gelatin and a colour-forming component with acid functional groups for a yellow, purple or azure image.

Examples of some colour-forming components for which the invention can be applied:

[structural formulae – see original]

Patent claim

A method of preparation of silver halide emulsions for colour photographic materials, characterised in that a salt of an alkaline earth is added in an amount of 0.02 to 2 g per 1 g of colour-forming component, at a temperature in the range 20-80°C, either to a solution of a colour-forming component with acid functional groups prior to incorporation in the emulsion, or to a photographic emulsion containing gelatin and a colour-forming component with acid functional groups.

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